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MEMORANDUM FOR PRS (In-House/Contractor Publication)

FROM: PROI (TI) (STINFO) 03 October 2000

SUBJECT: Authorization for Release of Technical Information, Control Number: **AFRL-PR-ED-TP-2000-189**
Vij, V.; Vij, A.; Wilson, W.W.; Christe, K.O. (ERC); Sheehy, J.; Boatz, J., Tham, F.S., "Chemistry of N-F Containing HEDM Materials and on Lewis Acidity of LiF"

HEDM Contractors Conference (Park City, UT, 24 Oct 2000) **(Statement A)**
(Deadline: 27 Sep 2000 – PAST DUE)

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Chemistry of N-F Containing HEDM Materials and on Lewis Acidity of LiF

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Abstract

The recent isolation of the N_5^+ cation is one of the most amazing discoveries of modern chemistry.[1] This statement is justified by the fact that this is the first all nitrogen homoleptic species to be isolated in more than a century, and only the third known species of this kind besides atmospheric nitrogen (N_2) and the azide ion (N_3^-). The $N_5^+AsF_6^-$ salt is only marginally stable at room temperature. In order to prepare other more stable N_5^+ salts for application as high energy density material, it was necessary to prepare the corresponding precursors in an efficient manner and in large quantities. The choice of common natural graphite flake as opposed to HOPG for the graphite. AsF_5 intercalate formation reduces not only the cost drastically, but also the intercalation time from several weeks to two days. This intercalate is then used to reduce N_2F_4 to N_2F_2. Using the graphite flake intercalate, this reduction reaction was accomplished in several hours instead of weeks with minimal formation of by-products. Another major discovery made during this study is the catalytic conversion of *trans*-N_2F_2 to its *cis*-isomer when SbF_5 is used as the Lewis acid. This reduces the number of steps required for the preparation of the precursor, $N_2F^+SbF_6^-$, and avoids the need for an extra step involving the corresponding expensive AsF_6^- salt. Also, discovered during the isomerization studies is a novel synthetic route to prepare NF_4^+ salts at relatively low temperature and pressure that does not require elemental fluorine and NF_3. A structural

analysis of the N_2F^+ cation was also carried out by the crystal structure determination of $N_2F^+SbF_6^-$ and $N_2F^+Sb_2F_{11}^-$. Isolated N≡N and N-F bond distances were measured for the first time in the solid state. These distances are in excellent agreement with those predicted by theoretical calculations. The N≡N bond in $N_2F^+Sb_2F_{11}^-$ is shorter than the triple bond in N_2 and is the shortest known nitrogen-nitrogen bond.

In addition to the structure and chemistry of N-F compounds, an attempt was also made to synthesize and characterize $CsLiF_2$ in order to study the isolated LiF_2^- anions. According to theoretical calculations and the recently developed pF^- scale, free gaseous LiF ($pF^- = 7.23$) is a much stronger Lewis acid than CsF ($pF^- = 3.8$) and, therefore, should easily abstract the fluoride ion from CsF with the formation of the LiF_2^- anion. However, the linear LiF_2^- anion, which is theoretically predicted to be vibrationally stable in the free gaseous state, could not be isolated. The single crystals obtained from a fused 1:1 eutectic mixture show that $CsLiF_2$ exhibits an interesting three-dimensional network of alternating tetra-coordinated lithium and octa-coordinated cesium cations bridged by fluorine atoms in a *C*-centered monoclinic lattice.

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1. C&E News, Jan 25, 1999; C&E News August 14, 2000, *and references therein.*